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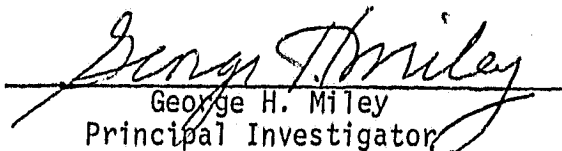
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Final Technical Report

NASA Grant NSG-1547

An Experimental Investigation of $^{235}\text{UF}_6$ Fission Produced Plasmas

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Abstract

A gas handling system capable of use with UF_6 was designed and constructed for use with nuclear pumped laser experiments with the University of Illinois TRIGA research reactor. By employing careful design and temperature controls, the UF_6 can be first transported into the irradiation chamber and then, at the conclusion of the experiment, returned to gas cylinders.

Table of Contents

	<u>Page</u>
I. Introduction	1
II. System Design.	6
References.	23
Appendix A: Operating Procedures for UF ₆ and Gas Handling Systems	24
Appendix B: Publications Issued Under Contract	26

An Experimental Investigation of $^{235}\text{UF}_6$ Fission Produced Plasmas

I. Introduction

Considerable interest exists in pumping nuclear-pumped lasers (NPLs) with the energetic fragments produced by fission of ^{235}U distributed throughout the laser gas as UF_6 vapor[1,2]. The quenching effect of UF_6 on gas lasers is unknown but is expected to be considerable. However, UF_6 mixtures can be used as a nuclear "flash lamp" to produce radiation for pumping a second laser mixture, and due to the high efficiency anticipated with this approach, it appears quite attractive.

The main objective of the present project was to design and construct a UF_6 handling system that could be used in conjunction with the existing NPL Group vacuum system at the Nuclear Reactor Laboratory to perform NPL experiments utilizing UF_6 . A secondary objective was to separate the gas fill system from the vacuum system and thus greatly reduce its volume, i.e., reduce the volume of expensive, ultra-pure rare gases (e.g., Xenon) needed in some NPL experiments.

Figure 1 is a phase diagram for UF_6 . As can be seen, UF_6 is a solid at room temperature. Additionally UF_6 contains copious amounts of F which is very corrosive. Finally, U (esp. ^{235}U) possession and usage is regulated by the NRC. For these reasons UF_6 presents special problems for the gas laser experimentalist.

Our usual gas handling method involves connecting high pressure bottles of the gases of interest to a manifold, through copper lines, and then through another copper, brass, or SS line to the laser, which is also connected to a vacuum system. The laser, manifold, and copper lines are pumped down to the

residual gas pressure desired, then the desired amounts of laser and buffer gases are added. This method is not satisfactory for UF_6 , obviously, since it is a solid at pressures above a couple hundred torr (at room temperature) and very corrosive.

Mercury and other metal vapor lasers face a somewhat similar problem. However, there are major differences. Nuclear-pumped lasers at the TRIGA reactor are $>.4$ m from any possible location for an oven vapor source and the vapor densities required are much greater, 10 - 100 torr vs. m torr. For Hg lasers, the oven problem was solved by putting small amounts of Hg in reservoirs within the laser envelope and the laser, which must be heated anyway, became the oven. This is unsatisfactory for UF_6 because the excess UF_6 would still react with neutrons if enriched. Also, the UF_6 could be cracked, producing $\text{UF}_x (x < 6)$ solids, or react with the fluorine bearing laser gases, contaminating all of the UF_6 in the laser. Only the precise amount of UF_6 required as vapor may be present in the laser at any given time. Therefore, in addition to heating and temperature control of the laser cell, a method is required to transport the UF_6 vapor over a distance of several meters from the UF_6 vapor source to the laser cell.

The method that we selected for UF_6 transport relies on the vapor pressure-temperature relationship shown in Figure 1. By imposing a temperature differential one also imposes a pressure differential causing the UF_6 vapor to be transported from the region of higher temperature and thus pressure to the region of lower temperature.[3] Such a transport system is practical only for a material which goes from solid to vapor (and back) with the attendant large changes in

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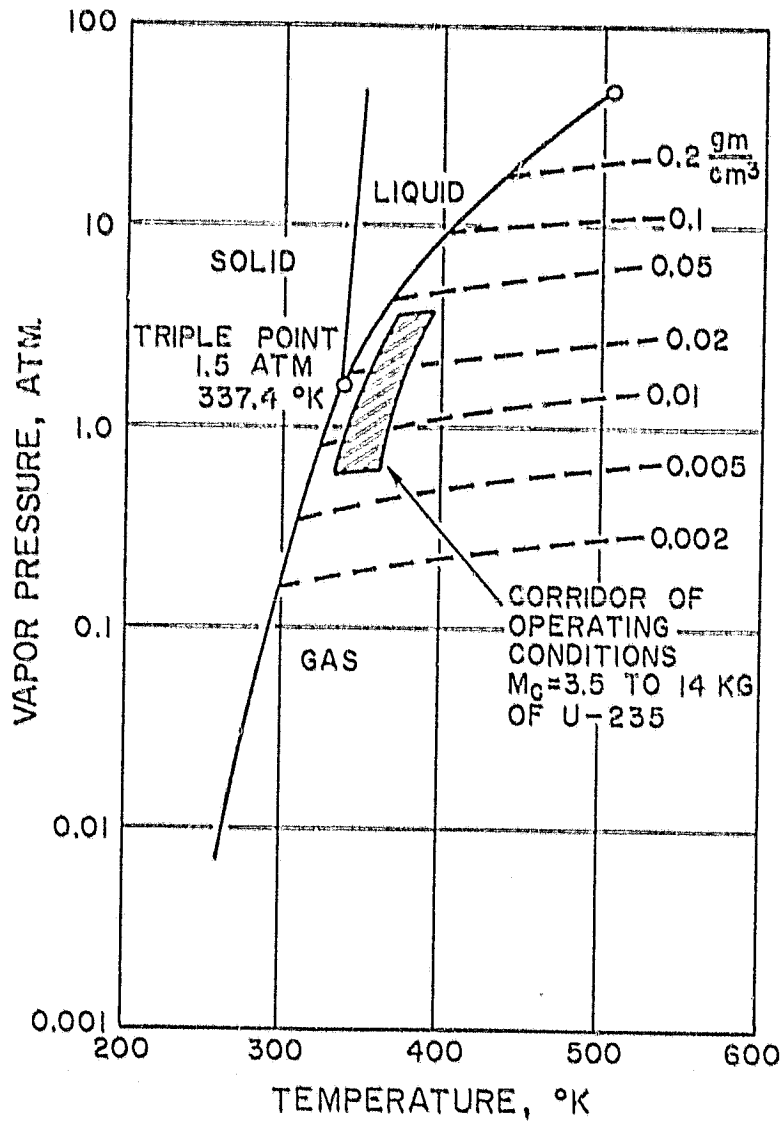


Figure 1 State Diagram for UF_6

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specific volume. Thus, the laser gases must be transported by the usual methods: filling the laser cell from a high pressure reservoir and evacuating the laser cell with a vacuum pump to the atmosphere. Since the UF_6 is to be returned to a spent UF_6 cylinder, rather than vented to the atmosphere, and because it would most likely badly mess up the vacuum system, the UF_6 must be separated from the laser gases.

Table 1 compares the boiling or sublimation temperatures for the laser gases of interest with that of UF_6 and CO_2 . It can be seen that a dry ice trap can be used to separate UF_6 from XeF^* laser gases. (But not from any byproduct HF that is formed. This can be removed in a separate operation at room temperature.)

Table 1
Boiling or Sublimation Temperatures of Interest
at 1 Atm.

<u>Substance</u>	<u>Temp (K)</u>
N_2	77
Ar	84
F_2	85
NF_3	144
Xe	166
CO_2	195
HF	293
UF_6	329

Figure 2 is a block diagram of the gas and UF_6 handling systems. The arrows indicate flow direction. A typical experiment is run as follows (a detailed operating procedure is given in Appendix A):

- (1) the laser cell is evacuated to the desired residual gas pressure (determined by allowed impurity concentration,
- (2) the laser is filled with the desired mixture of laser gases and UF_6 ,
- (3) the reactor is pulsed and data taken,
- (4) the "used" UF_6 is returned to the UF_6 supply system and the contaminated (with chemical reaction products) laser gas exhausted.
- (5) If it is desired to change the laser gas mixture for the next pulse, the gas fill line and manifold are evacuated.

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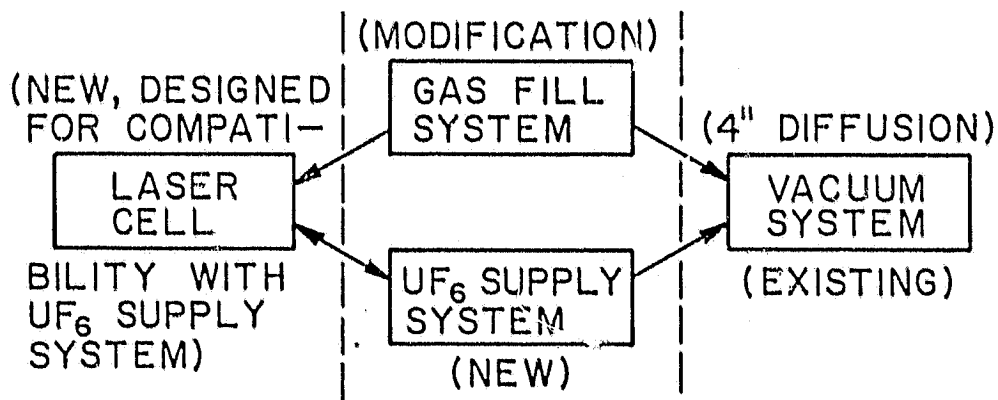


Figure 2 Block Diagram of Apparatus for
 UF_6 -NPL Experiments

II. System Design

The re-design of the gas fill system was basically trivial. The modified system design is shown in Figure 3. The modification involves the addition of the components to the left of the dotted line. A new valve is connected to the existing gas fill manifold. From this valve the laser gasses pass through a dry ice trap and a long, small diameter ($1/8'' \rightarrow 1/4''$ OD) SS tube to the laser cell.

The UF_6 Supply System design problem was somewhat more demanding. It is divided into two parts. The first part was the design of the vacuum envelope, which assumes the presence of a temperature control system; the second part was the design of the temperature control system.

II.A. Vacuum Envelope

Figure 4 is a schematic of the vacuum envelope of the UF_6 supply system. In this section, the details of the vacuum envelope will be considered. In the next section we will consider temperature control, shown cross-hatched in Figure 4.

Laser Cell

The most critical part of the vacuum envelope design is the laser cell where the actual experiment takes place and which is exposed to nuclear radiation. The internal details of the laser cell are set by laser physics considerations and will be discussed here only as they effect the vacuum envelope. One of these considerations is that, in addition to being compatible with fluorine compounds (NF_3 , UF_6) and neutron radiation, to permit the inclusion of electrical pumping the laser cell materials must be insulators. Teflon (TFE) and poly-vinyl chloride (PVC) were identified as suitable materials. As designed the cell uses PVC for tubing and TFE for parts machined from rod because they are most available in these forms. "Torrseal" low vapor pressure epoxy is used for attaching the PVC to the TFE. Fused silica, which maintains

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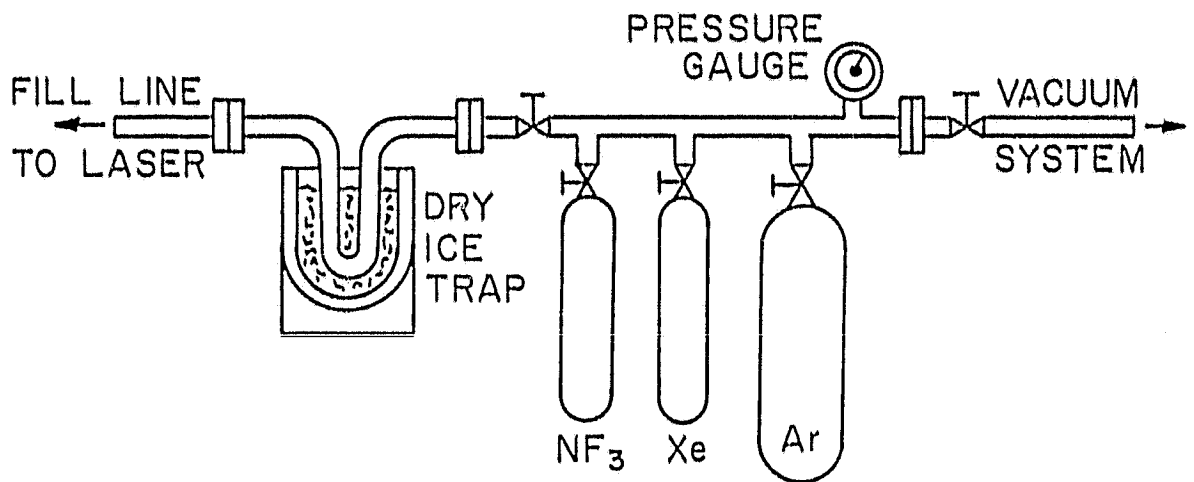
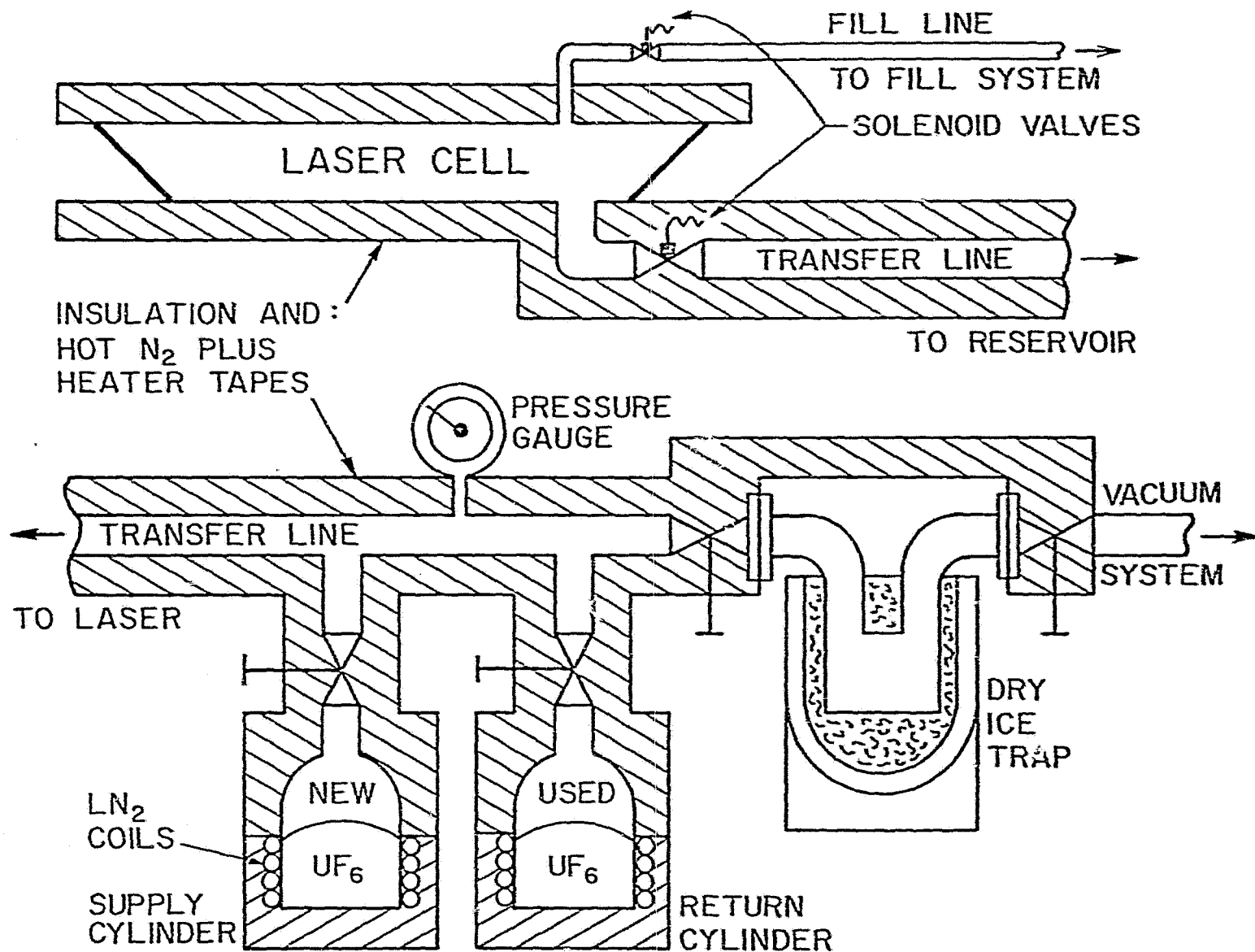


Figure 3 Gas Fill System

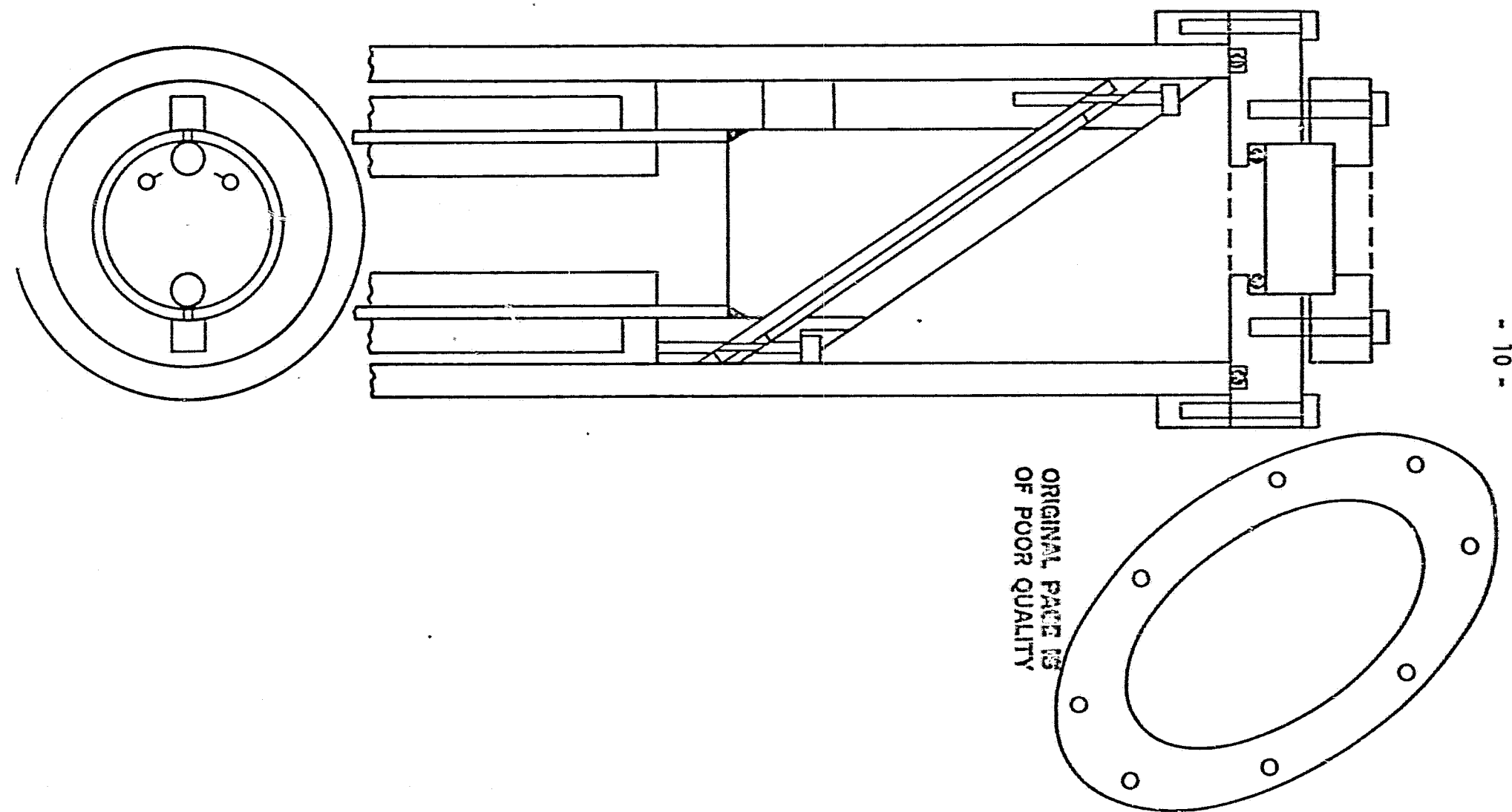
Figure 4 UF_6 Handling System Schematic

its >99% transmissivity under irradiation (because of its high purity) is used for windows, and viton is utilized for the gaskets. Figure 5 shows various views of the laser cell. A prototype cell has been constructed using acrylic in place of TFE.

Transfer Line

For a very long vacuum line (such as is required at the TRIGA) the ultimate pressure obtained at the end of the vacuum line (i.e. the laser cell) is dependent on the inverse of the cube of the diameter of the vacuum line ($\propto \frac{1}{d^3}$) and independent of the pump capability (unless the pumping speed is very low). [4] Thus it is desirable to make the effective diameter of the vacuum line as large as possible. However, limited space in the throughport makes a very large diameter line into the laser cell impossible. The diameter of the throughport is 6". A 1" ID cylindrical volume, concentric with the centerline of the throughport, is reserved for the output from the laser cell. Thus a 2 1/2" thick annular cross-sectional area is available for the laser cell envelope and the hardware for connecting the laser cell to the gas fill, vacuum, and UF_6 handling systems. As shown in Figure 6, the largest diameter circular cross-section transfer line that can be accommodated is 1"-1 1/4". Any geometry other than circular would probably be too expensive to fabricate and would not have a much more favorable surface area to volume ratio. The vacuum line currently used is 1" in dia. and residual gas concentrations have been found to be satisfactory.

The transfer line vacuum envelope was constructed of SS tube because of its availability (relative to Aluminum and Inconel).



- 10 -

Figure 5 Nuclear and Electrical Pumped "Hybrid Laser Cell
for UF_6 -RPL Experiments

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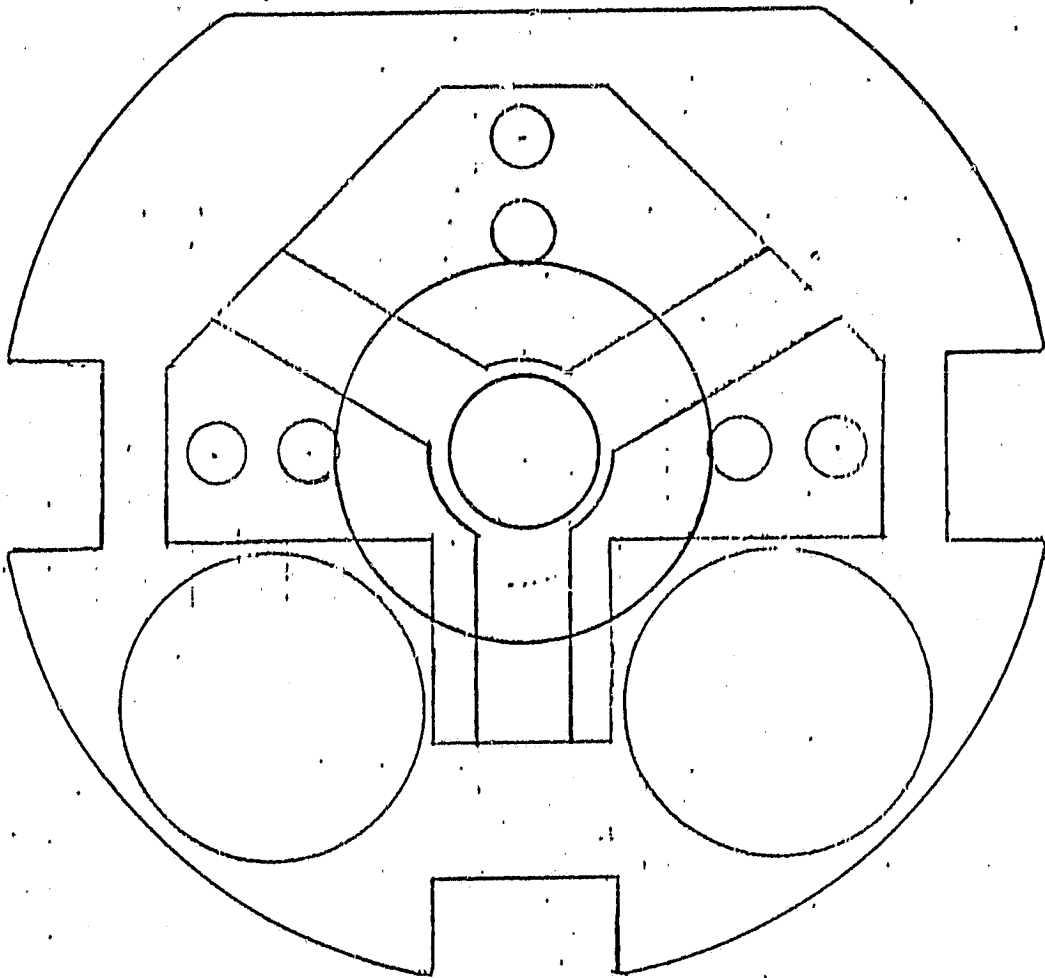


Figure 6 Diagram of Laser Carriage for TRIGA Reactor
Thru-port with Maximum Diameter Transfer
Lines Indicated (Full Scale)

UF₆ SUPPLY

As can be seen from Figure 1, any reasonable Δp desired can be achieved (by heating) for transporting UF₆ to the laser cell. This is not true for the reverse process. Also only a small fraction of the UF₆ contained in the supply cylinder must be transported to the laser cell. However, we wish to return essentially all of the UF₆ from the cell to the gas cylinder. It was not obvious initially that cryopumping the UF₆ would work or what size to make the system; but these problems were worked out as described below.

Reference 3 used four 1-l metal cylinders. The only "cryopumping rate" that they report is the return of 20(\pm .5)g of 25(\pm .5)g of UF₆ in one hour. However, their "canister" is 85l in volume vs. 1/2 l for our laser cell (~2 1/2 l for the transfer line) and the most UF₆ anticipated to be in the laser cell at one time is ~1 g. Thus, our pumpdown rate should be somewhat better.

The maximum cryopumping speed can be estimated by assuming that all UF₆ molecules impinging on the cold UF₆ in the return cylinder are trapped. The impingement rate is given by[4]

$$\gamma = \frac{1}{4} n \bar{v} = 3.5 \times 10^{22} \frac{p}{\sqrt{MT}} \quad (1)$$

The inside diameter of the cylinder can be taken as 6 cm. Thus, the pumping speed of the cylinder can be calculated from[4]

$$S_{\max} = \frac{Q}{p} = \frac{\gamma \cdot A \cdot kT}{p} = 3.6 \sqrt{\frac{T}{M}} \cdot A \quad (2)$$

Substituting the M value for UF₆, 350°K, and the area corresponding to a 6 cm diameter gives

$$S_{\max} \approx 100 \text{ l/s} .$$

This is a pumping speed comparable to a small diffusion pump.

Estimation of the pumpdown time to a given UF_6 density (or partial pressure) is more difficult because it requires knowledge of the surface properties of the various materials involved for UF_6 . However, several simplified approximations can be made.

The crudest approximation is to neglect surface effects (and the finite conductance of the transfer line) altogether. Thus we are essentially considering a 3l volume with an $\sim 30^2$ cyropumping area. From reference 4,

$$\frac{dN_{\text{UF}_6}}{dt} = -\gamma A = -\sqrt{\frac{8kT}{\pi M_{\text{UF}_6}}} \frac{\dot{N}_{\text{UF}_6}}{V_{\text{system}}} \cdot A_{\text{cyl}} \quad (3)$$

or

$$\frac{dN_{\text{UF}_6}}{N_{\text{UF}_6}} = -\sqrt{\frac{kNA}{2\pi}} \sqrt{\frac{T}{M_{\text{UF}_6}}} \frac{A_{\text{cyl}}}{V_{\text{system}}} dt = -36.4 \sqrt{\frac{T}{M_{\text{UF}_6}}} dt . \quad (4)$$

For $T = 350\text{K}$,

$$\frac{dN_{\text{UF}_6}}{N_{\text{UF}_6}} = -36.4 dt$$

so that

$$\frac{N_{UF_6}}{N_{UF_6}(0)} = e^{-36.4t}$$

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(5)

For $\frac{N}{N_0} = 10^{-8}$, t is about .5 sec.

While this result is very crude, pumpdown times more than three orders of magnitude greater would be acceptable.

Another crude approximation is to treat the system as a normal vacuum system made of materials with high outgassing rates.

Taking $S_{eff} \approx S_{th} = S_{max}$, assuming that $\frac{S_{max}}{L} \ll |e|$ and molecular flow regime, and neglecting $p_{ult.}$; the pumpdown time is given by [4]

$$t = \frac{V}{S_{max}} \ln \left(\frac{p_i - \frac{Q_g}{S_{max}}}{p_f - \frac{Q_g}{S_{max}}} \right) \quad (6)$$

It has previously been determined that V is ~ 3 l and S_{max} is ~ 100 l/s.

$Q_g = q_g \cdot A_{system}$ and q_g can be approximated by taking a high value of 10^{-5} (torr-liter)/(S-cm²)[4]. Then

$$Q_g = 10^{-5} \frac{\text{torr-liter}}{\text{sec-cm}^2} \cdot 4 \times 10^3 \text{ cm}^2 = 4 \times 10^{-2} \frac{\text{torr-liter}}{\text{sec}}$$

This says that the lowest attainable pressure is

$$P_{lowest} = \frac{Q_g}{S_{max}} = \frac{4 \times 10^{-2} \frac{\text{torr-liter}}{\text{sec}}}{100 \frac{\text{liter}}{\text{sec}}} = 4 \times 10^{-4} \text{ torr.}$$

An infinite amount of time would be required to achieve this pressure. Using equation (6), the time to pump down from 100 torr to 5×10^{-4} torr would be

$$t \approx \frac{3 \text{ l}}{100 \text{ l/s}} \ln \frac{100}{5 \times 10^{-4} - 4 \times 10^{-4}} \approx .4 \text{ sec} .$$

Again it appears that pumpdown times will be very satisfactory. And, while a UF_6 pressure of $\sim 5 \times 10^{-4}$ torr is not ideal, it represents about 10 μg for our system volume -- a negligible amount, even for NPLs or the NRC.

The result of these crude calculations and the comparison to reference 3 is that it appears that cryopumping using a single 1-l UF_6 cylinder will satisfy the needs of the experiment quite well. Indeed, this has been found to be the case.

II.B. Temperature Control

Since the UF_6 transfer system utilizes a vapor pressure pumping scheme (the return portion of the cycle being the well known cryopumping process), accurate control of temperature is important. Additionally, uniformity of temperature is important since having the temperature drop below the sublimating temperature at any point would result in the deposition of UF_6 at that point. This is of special concern for the sealing surface of the valves.

We have designed a temperature control system that attempts to provide maximum performance at minimum cost. Factors included in performance include accuracy of temperature control at a point, uniformity of temperatures, flexibility, and minimum of manual intervention. The temperature control system can be divided into two basic subgroups:

- (1) Heating and cooling
- (2) Temperature sensing and control electronics

1. Heating and Cooling

Four different types of hardware must be heated. These are: (a) the laser cell, (b) the transfer line, (c) the UF_6 cylinders, and (d) valves.

(a) Laser Cell Heating and Insulation

The laser cell, Figure 5, it is not very conducive to the use of heater tapes since they would have to be applied to the outside of the capacitor. The capacitor and the "air" gap between the capacitor and the laser cell provide a considerable thermal resistance. As will be explained in the temperature sensing section, thermistors are the desired method of temperature sensing. However, the lifetime of thermistors in the severe radiation environment of the reactor is unknown and probably very short. Both of these problems can be solved by the use of flowing hot nitrogen

heating. The hot nitrogen can fill the gap between the capacitor and the laser cell and by flowing it, temperature sensing can be moved outside the reactor. Nitrogen is chosen over air because it is not reactive with UF_6 or any of the laser gases. By maintaining an N_2 pressure higher than the pressure in the laser, any leakage will be of N_2 into the laser.

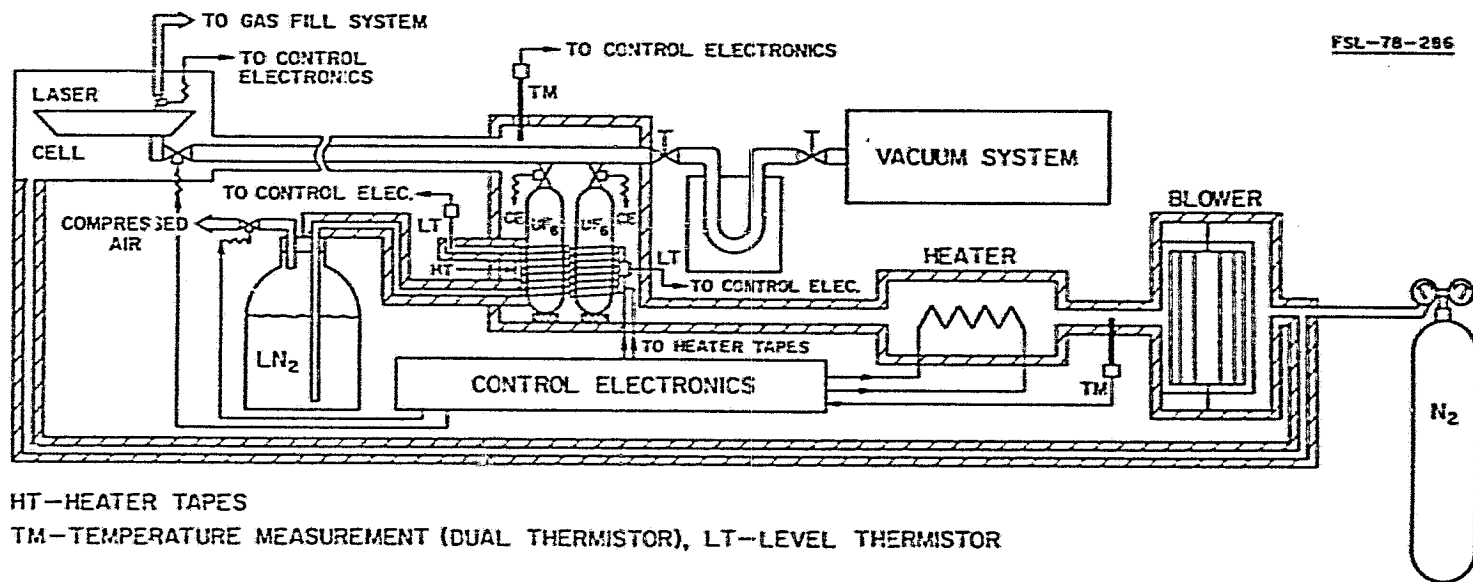
(b) The transfer line could be heated by either the hot N_2 used for the laser cell or by heater tapes as with the valves. Both methods have advantages. Heater tapes are very easy to install, one simply wraps them around the transfer lines, and occupy a minimum volume (if no further insulation is used), which can enable the maximization of the cross-sectional area of the transfer line (the importance of which was discussed earlier). On the other hand, with heater tapes, one controls the heating rate rather than the temperature so one must accurately measure the temperature. However since one end of the transfer line is in a high radiation environment thermistors cannot be used. Thermocouples are much less accurate. Heating and temperature measurement would probably be required in a minimum of three regions, the middle and the two ends, making the control problem slightly more complex than it could be. Similarly, six pairs of wires would be required.

Since hot N_2 is already required for the laser cell, no additional hardware is required for its use with the transfer line other than the co-axial envelope around the transfer line required to contain the temperature can be maintained more accurately and uniformly than with heater tapes. The two disadvantages are 1) that any co-axial flow design will be bulkier than heater tapes without insulation and 2) a 4-5 m co-axial flow line will be relatively complex and expensive to build.

In summary, a heater tape system is simpler to construct but more complicated to operate and allows a greater conductance (and thus vacuum system) performance) but gives poorer temperature control performance than a co-axial flow system. Figure 7 depicts the hot N_2 approach which we have chosen.

(c) The UF_6 cylinders require both heating and cooling: heating for vaporizing the UF_6 and generating the desired pressure to transport it ~5 m to the laser cell and cooling, with liquid nitrogen, to cryopump the UF_6 back. In fact, for the cryopumping phase, heating and cooling are required simultaneously - the inlet to the cylinder will be heated to prevent plugging. As shown in Figures 3 and 7, copper cooling coils are soldered to the bottom half of the monel UF_6 cylinders. For cooling, LN_2 is run through the copper coils. Heating is provided by a combination of heater tapes and hot N_2 . The cylinders and valves are wrapped with heater tapes, in two regions, and inserted in an airtight, insulated enclosure connected to the hot N_2 flow system. Any UF_6 leaks flow into this enclosure.

(d) Five valves require heating. These are the two UF_6 cylinder valves, the laser cell to transfer line valves, the transfer line to transfer line trap valve, and the transferline trap to vacuum system valve. All of these are heated using heater tapes. The heater tapes are wrapped around the valves and fiberglass insulation are warpped around the heater tapes.



HT-HEATER TAPES

TM-TEMPERATURE MEASUREMENT (DUAL THERMISTOR), LT-LEVEL THERMISTOR

Figure 7 Temperature Control System for UF_6 Handling System

2. Temperature Sensing and Control Electronics

The temperature of greatest interest is that in the laser cell. This will be measured using one or both of two methods. The preferable method depends on the effectiveness of a shielding material called flex boron which has recently come to our attention (the NPL Group) but which we have yet to test in the reactor. If effective, we are able to use it to shield a number of neutron radiation sensitive components (the unknown is the fraction of current damage caused by γ 's), including thermistors, one of which is located in a shielded portion of the laser cell. In either case, the inlet and outlet N_2 temperatures are measured using thermistors and the cell temperature inferred from these N_2 temperatures. Similar thermistors are used to measure the temperature in the UF_6 supply enclosure.

The electronics for the thermistor temperature measuring system are shown in block diagram form in Figure 8. Such systems are highly accurate even though relatively inexpensive. A similar system is used by the Combustion Engineering BoronometerTM (designed by the author). With a 10 KHz V/F and a 1 second counting time, resolution was $.01^{\circ}C$ and error was $<.1^{\circ}C$. For this system an $\sim.1$ second counting period is used to produce a resolution of $.1^{\circ}C$ and an error of $\sim.1^{\circ}C$. The thermistor that was used is a unique dual thermistor (manufactured by Yellow Springs Inst.) that has an output that changes linearly with temperature. A monolithic instrument amp (such as the Analog Devices 521) raises the voltage level of the thermistor output so that the maximum design temperature equals the full scale voltage of the voltage to frequency convertor (V/F), which functions as an analog to serial-digital converter. The serial-digital data is transmitted to a microcomputer where it is converted to temperature units and displayed or used for control.

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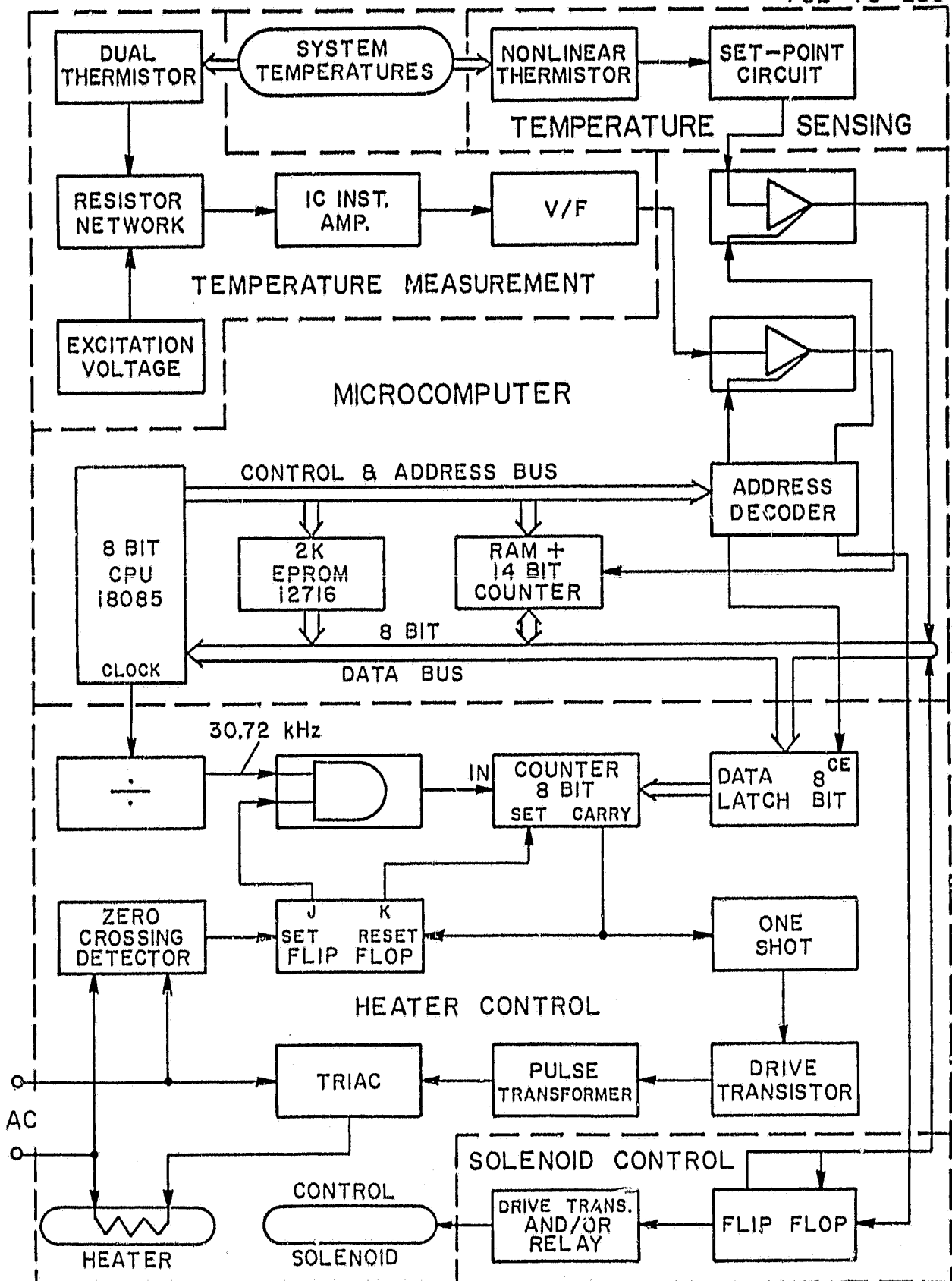


Figure 8 Temperature Sensing and Control Electronics for UF_6 Handling System

Items that will be controlled include the temperature and flow rate of the hot N_2 , the flow of the LN_2 , and the various system solenoid valves.

The solenoid valves are the easiest to control. The μ -computer address is decoded to drive a transistor which drives the solenoid directly or through a relay. The LN_2 flow is controlled in a similar manner using air pressure controlled by a solenoid to propel the LN_2 . (This method is used for keeping the diffusion pump cold trap filled in the vacuum system currently in use at the reactor.)

A somewhat more complex system is used to control the N_2 heater and the heater tapes. According to the heating rate desired, the μ -computer outputs an eight bit digital word, corresponding to the fraction of the 60 Hz power frequency phase required to produce that heating rate, to a latch. At the start of each 60 Hz half-cycle a counter is reset from the latch and counted down to zero at which point it produces a pulse which turns on the triac and allows power to flow in the heater circuit for the remainder of the half cycle. If the word is 0 power starts flowing immediately. If the word is 256 the triac will never turn on. Precision of control will be better than 1 part in 100.

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Appendix A

Operating Procedures for UF_6 and Gas Handling Systems

for NPL- UF_6 Pumping Experiments

A. System Preparation (1-5 performed only at the beginning of each series of experiments)

1. Open gas and UF_6 handling systems and laser cell to vacuum system, pump down and bake out (with diffusion pump).
2. Valve-off diffusion pump, chill UF_6 cyl. and transfer line trap to 0°C (ice bath, after initial cooling with LN_2), fill with inert gas, and open UF_6 valves. Allow accumulated HF to be pumped out (note: forepump is exhausted through reactor stack).
3. Close valve between transfer line trap and vacuum system. Chill UF_6 supply cyl. with LN_2 and then the heat trap with a heat gun, thereby cryopumping UF_6 from trap into cylinder.
4. Cool transfer line trap and gas fill line trap with dry ice.
5. Close-off cylinders from transfer line and open transfer line trap - vacuum system valve.
6. Open diffusion pump valves and pump down system to desired background pressure.

B. Transfer UF_6 from supply cyl. to laser cell.

1. Heat supply cyl., transfer line, and laser cell to desired temps.: laser cell and transfer line to $T_{\text{subl}} + 25\text{K}$ (for desired n_{UF_6}), supply cyl. to temp. which will provide a P_{sat} which, when expanded into laser cell, will provide the desired n_{UF_6} . Start cooling UF_6 return cyl. with LN_2 .
2. When desired temps. are achieved, close laser cell to gas fill line valve and transfer line to transfer line trap valve (thus valving UF_6 system and laser cell off from vacuum system).
3. (a) Set temp. control on supply cyl. to provide temp. for sat. pressure for desired n_{UF_6} , then
(b) open UF_6 supply cyl. valve. [Expansion of UF_6 into transfer line and laser will cool UF_6 , $T = T_0 \frac{V_0}{V} \gamma - 1$, $q = C_p/C_v$].
4. Once pressures and temperatures stabilize: (a) close transfer line valve to laser, (b) start cooling supply cyl. in order to cryopump extra uncontaminated UF_6 in transfer line back into supply cyl. and, (c) start cooling transfer line trap with dry ice.
While supply cyl. is cooling and pumping go on to...

C. Fill laser cell with laser and buffer gases

1. (a) Close gas fill manifold valve to vacuum system.
(b) For laser systems with small atom fraction constituents (.1%) also close manifold - gas fill line valve.
2. (a) Add gases to manifold or gas fill line in order of increasing atom fraction (add to desired pressure times volume factor).
(b) If small atom fraction constituents, add these plus portion of buffer gas first to manifold only (using volume factor of manifold to manifold + fill line + laser cell) then expand into manifold plus fill line and add remainder of buffer gas to desired pressure (taking into account volume factor).
3. Open valve between gas fill line and laser cell and, as soon as pressure fluctuations subside, close valve again. [Since UF_6 pressures will be <20% of total pressure in laser cell and since volume factors should be 2 or greater, pressure in the fill line should be 10-100 times greater than the UF_6 pressure in the laser cell so that UF_6 loss from the laser cell into the gas fill line should be minimal - hopefully negligible.]
4. After allowing time for temperatures to stabilize and for gas mixing by diffusion, the laser cell is ready for pulsing.

D. Pulse reactor and take data (covered by separate procedure)

E. Evacuate gases and return UF_6 to return cyl.

1. Check to see that transfer line trap is cold (dry ice cooling)
2. Shut UF_6 supply cylinder valve and diffusion pump valve
3. Open transfer line - transfer line trap valve and pump out gases [UF_6 is caught in trap] to best pressure available with forepump.
4. Close transfer line trap - vacuum system valve and open UF_6 return cylinder valve.
5. Remove cooling from transfer line trap and heat with heat gun until all UF_6 and other cryopumpable residuals are returned to return cylinder.
6. Close return cylinder valve. Open vacuum system to transfer line trap and laser cell to gas fill valves.

System is now ready to return to A.6 and recycle.

Appendix B

Publications Issued Under Contract

- UPL-01 "Production of XeF*(B) by Nuclear Pumping"
(G. H. Miley, F. P. Boody, S. J. S. Nagalingam, and M. A. Prelas, *Thirty-First Annual Gaseous Electronics Conference*, BA-7, p. 32, Buffalo, NY, 17-20 October 1978)
- UPL-02 "Recent Results with the Atomic Carbon Laser @ 1.4539 m"
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